THE NICKEL BULLETIN

A SUMMARY OF [illegible] INFORMATION ON NICKEL

MAY, 1953

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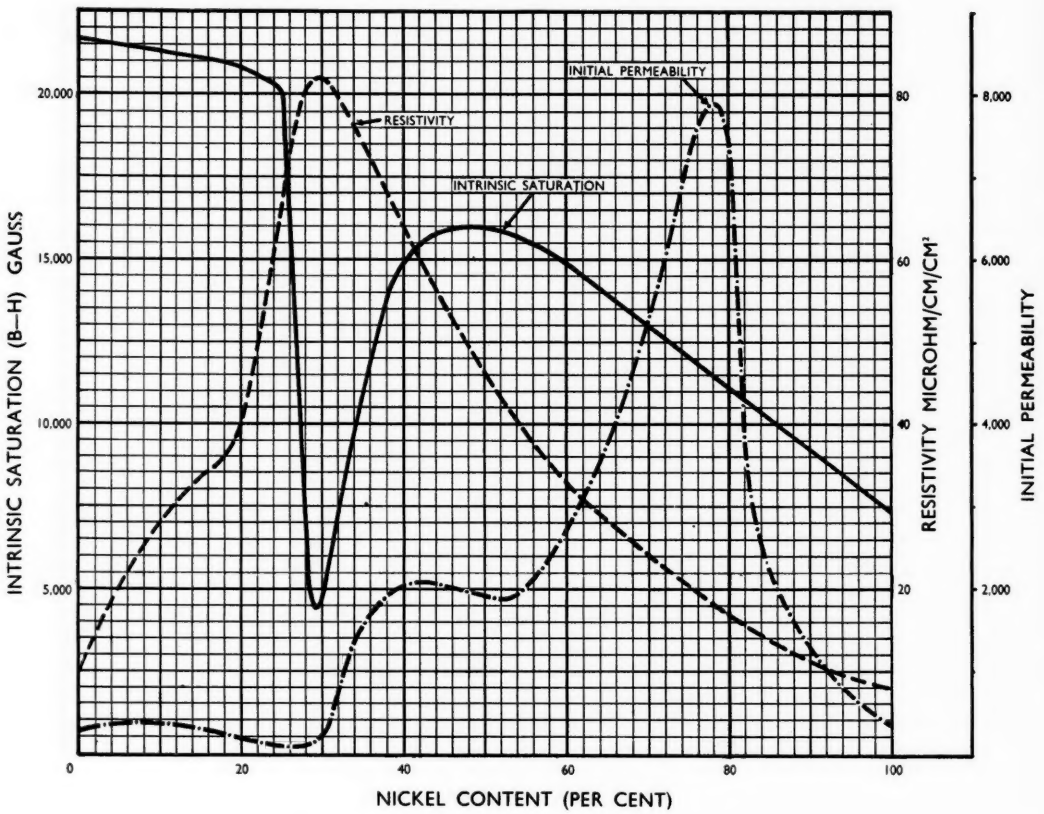
VOLUME 26 NUMBER 5

THE MOND NICKEL COMPANY LTD • SUNDERLAND HOUSE, CURZON STREET, LONDON, W.1

Telephone:- Grosvenor 4131

MAGNETIC PROPERTIES OF THE NICKEL-IRON ALLOYS

The remarkable magnetic properties of certain nickel-iron alloys, and in particular their high permeability and low hysteresis losses at low field strengths, have led to widespread application in telecommunication and in light electrical engineering; the more important uses include small transformers, magnetic screens, sensitive relay and pick-up cores, etc., etc.



Fundamental Properties of the Nickel-Iron Series

Curves for Intrinsic Saturation and Resistivity taken from T. D. YENSEN: 'Magnetic and Electrical Properties of Nickel-Iron Alloys.' *Jnl. Amer. Inst. Elect. Eng.*, 1920, vol. 39, pp. 396-405.

Curve for Initial Permeability taken from W. G. ELMEN: 'Magnetic Alloys of Iron, Nickel and Cobalt.' *Jnl. Franklin Inst.*, 1929, vol. 207, pp. 583-617.

Modifications of the nickel-iron alloys, involving minor additions of other elements, special processing, etc., have led to the production of improved materials with still better magnetic properties. These materials can conveniently be divided into three main classes containing approximately 78, 50 and 35 per cent. nickel respectively. The first of these (A) is characterised by the highest permeability but with a relatively low saturation value; in the second and third classes (B) and (C), whilst still maintaining relatively high permeabilities, the saturation and resistivity have been respectively increased. Special properties have also been developed in certain alloys by means of orientation of metallurgical structure (Class D).

Typical Data for British Alloys

		CLASS A				CLASS B				CLASS C				CLASS D	
		Permalloy C		Mumetal		Permalloy B		Radiometal		Permalloy D		Rhometal		HCR	Permalloy F
NICKEL CONTENT (approx. per cent.)		77		77		45		45		36		36		(oriented structure)	
INITIAL PERMEABILITY		20,000		28,000		2,000		2,000		1,700		1,500		1,000	1,000
SATURATION INDUCTION gauss		8,000		7,000		16,000		16,000		13,000		9,000		16,000	14,000
MAXIMUM PERMEABILITY at Field Strength H in Oersteds		80,000 0·03		90,000 0·03		15,000 0·4		20,000 0·3		7,000 0·7		8,000 0·5		60,000 0·2	300,000 0·05
ELECTRICAL RESISTIVITY microhms/cm/cm²		60		60		55		55		90		95		45	26
SPECIFIC GRAVITY		8·6		8·8		8·3		8·3		8·15		8·1		8·25	8·4
MECHANICAL PROPERTIES	*Condition*	*Cold rolled*	*Annealed*	*Cold rolled*	*Annealed*	*Cold rolled*	*Annealed*	*Cold rolled*	*Annealed*	*Cold rolled*	*Annealed*	*Cold rolled*	*Annealed*		
	MAXIMUM STRESS tons/sq.in.	65	43	60	35	65	35	45	25	33	25	65	35		
	ELONGATION per cent.	0·5	30	1	30	0·2	20	0·7	25	20	30	0·5	30		
	HARDNESS D.P.N.	310	120	290	100	220	110	240	100	140	130	290	110		

The following, amongst others, are trade names used for alloys produced abroad:—

CLASS (A) (Highest permeability)	U.S.A.:	4-79 Permalloy & Supermalloy, Hymu.
	France:	Anhyster M.
	Germany:	1040 alloy, Megaperm 6510.
CLASS (B) (Highest saturation)	U.S.A.:	45 Permalloy, Hipernik, Nicaloi, Allegheny 4750.
	France:	Anhyster D.
	Germany:	Permenorm, Hyperm, Megaperm 4510.
CLASS (C) (High resistivity)	France:	Anhyster B.
	Germany:	Isoperm.
CLASS (D) Oriented structure	U.S.A.:	Deltamax, Orthonol, Permeron.
	Germany:	Permenorm 5000Z.

ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

NICKEL

Nickel Production, 1952.

J. C. BROWNING: 'Ontario Annual Review, 1952.' *Canad. Mining Jnl.*, 1953, vol. 74, Feb., pp. 94-100.

This chapter is a section of the 1952 Annual Review of Canada's Mineral Industries. The portion relating to nickel is concerned mainly with expansion programmes of The International Nickel Company of Canada, Limited and of Falconbridge Nickel Mines. International Nickel has virtually completed its five-year plan, which will assure maintenance of the current yearly rate of 250,000,000 pounds of refined nickel. United States agencies have provided financial support to Falconbridge, making it possible for that Company to raise annual production to 35,000,000 pounds by the end of 1954. Some details are given of new ore bodies being exploited by these Companies, of smaller nickel mines which are being developed, and of some disused mines which are being re-opened.

See also—

'Ferroalloy Metals in Review: Nickel.' *Engineering and Mining Jnl.*, 1953, vol. 154, Feb., p. 98.

Section of 84th Annual Survey and Outlook Number. Notes on 1952 output from Canada, and of nickel oxide from Cuba; also nickel consumption, imports, and exports figures for the U.S.A. in 1952.

Mining of Nickel-containing Ores in the Sudbury Region: Inco Mines

H. J. MUTZ, A. F. BROCK and W. J. TAYLOR: 'Underground Mining Methods at International Nickel Company.' *Mining Engineering*, 1953, vol. 5, Jan., Sect. 1, pp. 57-82; T.P. 3461A.

Description of layout and methods used at the five underground mines worked by International Nickel Company of Canada, Ltd. (Frood-Stobie, Creighton, Murray, Garson, and Levack). At present open-pit mining is also in operation (on the surface ores of the Frood-Stobie orebody), but conversion to all-underground mining will be completed within the next two years, when the underground mines will have an annual hoisting capacity of 13,000,000 tons.

Methods of mining now employed in the Inco mines are square-set, cut-and-fill, shrinkage, blasthole, and induced caving. Choice of stoping methods is determined by conditions peculiar not only to the individual mine, but also to particular areas of any one mine. An account is given in this paper of the geology of the district, the considerations involved in selecting methods of stoping in the different areas, the general layout of the mines, and their equipment. The paper is illustrated by over 40 diagrams and photographs.

Behaviour of Evaporated Nickel Films on Annealing

R. W. HOFFMAN, F. J. ANDERS and E. C. CRITTENDEN: 'Evidence for Collapse of Lattice Vacancy Aggregates to Form Dislocation Rings.' *Jnl. Applied Physics*, 1953, vol. 24, Feb., pp. 231-2.

Seitz has proposed that, when the density of lattice vacancies exceeds the equilibrium value, the vacancies will coalesce into progressively larger groups, until they finally organize into sheets and collapse to form dislocation rings. Preliminary evidence, based on indirect measurement of the volume change occurring in nickel films during annealing at progressively higher temperatures, indicates the existence of the above mechanism. The conclusions are based on measurement of tensile stress in the films (as evaporated, and after annealing at various temperatures up to about 360°C.), electrical resistance of the nickel films as a function of annealing temperature, and a critical consideration of data recorded by other investigators relating to hardness, X-ray diffraction line breadth and other properties, as affected by annealing. The limited applicability of data obtained from evaporated films to bulk-metal specimens is conceded, but the authors are of the opinion that, in the present case, observations made on the film nickel may be interpreted as valid for bulk metal.

Hall Effect in Nickel and Nickel-Copper Alloys

See abstract on p. 80.

Measurement of Magnetostriction in Nickel and Nickel-Iron Alloy

See abstract on p. 81.

Oxidation of Nickel and Other Metals: Properties of the Oxides Formed

A. DRAVNIEKS: 'Correlations between Parabolic Oxidation of Metals and Properties of Oxides.' *Jnl. Electrochemical Soc.*, 1953, vol. 100, Mar., pp. 95-102.

From a search of the literature on parabolic formation of oxides, and from supplementary experimental work on oxidation of copper, nickel, and cobalt, kinetic constants are correlated with some physical properties of oxides. The paper is supported by a *bibliography* of 126 items.

Pickling of Metals and Alloys

See abstract on p. 80.

Welded Nickel Tubing for Transport of Caustic Soda

'Piping Hot Caustic Soda.' *Steel*, 1953, vol. 132, Jan. 26, p. 92.

This is a brief illustrated note on a 10,000-ft. long wrought-nickel piping system designed to carry caustic soda solutions at temperatures of the order of 600°F. (315°C.). In the course of fabrication, more than

2700 ft. of Heliarc welding has been done. The piping and fittings vary in diameter from 1¼ to 4 in., with wall thicknesses ranging from ⅛ to 3·8 in. Edge preparation for the joints varies. Some joints are hand ground to a 45° included angle, others to a sharper angle, with a 1/32 in. root face. Two passes are used on all joints: the first is made without a filler rod, in order to obtain a uniform underbead, and in the second pass a nickel welding rod is used. The current is d.c., straight polarity, about 100 amp. An argon gas-flow of approximately 10 litres per minute is used. The excellent resistance of nickel to corrosion by caustic makes this type of piping ideal for the purpose: even at the elevated temperature involved the attack is at the rate of less than 0·020 in. per year.

Method of Studying Basic Salts of Nickel

W. J. SINGLEY and J. T. CARRIEL: 'A New Method of Studying Metal Salts Applied to Certain Basic Salts of Nickel.' *Jnl. Amer. Chemical Soc.*, 1953, vol. 75, Feb. 20, pp. 778-81.

Following a condensed summary of the various experimental methods which have been used to investigate the composition and structure of precipitated basic metal salts, an account is given of a study of the factors influencing the formation of the basic salts of nickel. The work was done in connexion with preparation of high-purity nickel hydroxide, with the specific aim of avoiding formation of basic salts.

Standardized nickel-salt and potassium-hydroxide solutions were added from burettes to an alkaline medium, so that the pH of the latter remained constant. The composition of the precipitate was immediately calculable from the burette readings. Application of this method to an investigation of freshly precipitated basic nickel sulphates, chlorides, and nitrates showed that their composition was determined by the pH of the reaction medium. These basic nickel salts are metastable: their composition approaches that of nickel hydroxide at rates which are influenced by the temperature, the pH, and the neutral-salt content of the reaction medium. The authors were unable to find a satisfactory explanation for the observations made.

ELECTRODEPOSITION AND OTHER COATING METHODS

German Specification for Nickel-Plating Salts

DEUTSCHER NORMENAUSSCHUSS: 'Nickel Salts for Plating Baths.' *D.I.N. Specification* 50 970; issued as tentative Nov., 1952; comments accepted up to Apr. 30, 1953.

Attention is directed to the importance of using high-purity salts in plating, and to the necessity of ensuring dryness of nickel sulphate as delivered. The test proposed requires that the suitability of the salts be assessed by the quality of a deposit of 10μ thickness made, at a current density of 3 amp./sq. dm. and 40-45°C., on 0·5-mm. thick highly polished brass sheet, from a bath of the following composition:—

	g./L.
$NiSO_4.6H_2O$	240
$NiCl_2.6H_2O$	45
H_3BO_3	30
pH	4·5-5·5

It is also required that the brass sheet on which the deposit is made shall be so thoroughly degreased that on immersion in a nickel-plating bath without current on, and subsequent rinsing in water, there shall be no evidence of greasy patches on the surface. This test is to be made in a bath which has already been used.

The deposit is required to be matte or slightly bright, and it is specified that it shall not flake off when the plated strip is bent through an angle of 180°.

The following limits of composition are specified:—

	Nickel (II) Sulphate %	Nickel (II) Chloride %
Nickel + Cobalt	>20·6	>23·7
Cobalt	<2·0	
Iron	<0·05	
Copper	<0·01	
Zinc	<0·01	
Lead	<0·02	
Cadmium(1)	<0·02	
Free acid, calculated as sulphuric or hydrochloric acid ..	<0·1	
Constituents insoluble in sulphuric acid of pH=1	<0·2	

(1) In nickel salts for bright-plating baths the limit is <0·01 per cent.

Electrodeposition of Tin-Nickel Alloys from Fluoborate Baths

A. E. DAVIES, R. M. ANGLES and J. W. CUTHBERTSON: 'Complex Fluorides for the Deposition of Tin and Tin Alloys.' *Trans. Inst. Metal Finishing*, 1953, vol. 29, Advance Copy 7, Apr., 1953; 14 pp.

This paper describes some of the work carried out subsequent to a paper published by PARKINSON in 1950, on deposition of tin from fluoborate solutions. It reports experiments on (1) electrodeposition of tin from stannous fluoborate and stannous fluosilicate baths; (2) electrodeposition of tin-nickel, tin-cadmium and tin-zinc alloys from fluoborate baths, and (3) electrodeposition of tin-cadmium and tin-zinc alloys from fluosilicate baths. Although the work is in the relatively early stages, the authors are of the opinion that the types of bath studied are potentially useful: (1) fluoborate solutions may make possible increase in rate of plating; (2) they have been claimed to be superior to other electrolytes with regard to cathode efficiency; (3) they are relatively non-corrosive and can often be operated at room temperature; (4) they have proved promising in relation to electrodeposition of alloys.

Study of the possibility of using a fluoborate electrolyte as an alternative to the chloride-fluoride bath at present employed for deposition of tin-nickel alloys (see *Jnl. Electrodepositors' Tech. Soc.*, 1951, vol. 27, p. 129) showed that tin and nickel could be co-deposited from a mixed fluoborate bath, but that for the best results the presence of the fluoride ion is

desirable. A bath of the following composition has proved particularly suitable:—

	g./L.
Tin (as stannous fluoborate)	25
Nickel (as nickel fluoborate)	60
Ammonium fluoride	60

Working temperature 65° C.

The following information is given with regard to the influence of variables in tin-nickel alloy deposition from this type of bath:—

The stannous tin may be varied over the range 10-30 g./L., and the nickel over the range 20-100 g./L., without significant change in the composition of the plate.

As in the case of the chloride bath, the fluoride concentration is related to the stannous tin concentration, and the ratio of fluoride to stannous tin should be kept more or less constant. Sodium fluoride may be substituted for ammonium fluoride, if desired.

The brightest plate is obtained in the pH range 3·0-5·0. As the pH is lowered from 5·0 towards 3·0 the plate becomes somewhat brighter, but below pH 3·0 the deposit tends to become dull and the cathode efficiency falls.

Like the chloride solution, the fluoborate solution can be operated satisfactorily at cathode current densities up to 25 amp./sq. ft. If the solution is diluted good deposits can still be obtained, but only if the current density is reduced.

It is stated that up to the present the fluoborate bath for deposition of tin-nickel alloys cannot be claimed to offer any marked advantage over the established chloride-fluoride bath, but it is considered to be too early to draw final conclusions or predict possible future developments. Although the relatively high cost of fluoborates is an economic disadvantage, there is some benefit in the fact that the fluoborate solution is easier than the chloride-fluoride type to filter and keep clean.

Deposition of tin-nickel from the mixed fluoborate bath, the deposition of tin-zinc from the mixed fluoborate bath, and the deposition of tin-cadmium and of tin-zinc from mixed fluosilicates are the subject of three respective patent applications.

See also—

J. W. CUTHBERTSON, N. PARKINSON and H. P. ROOKSBY: 'Electrodeposition of Tin-Nickel Alloy Plate from Chloride-Fluoride Electrolytes.'
Jnl. Electrochemical Soc., 1953, vol. 100, Mar., pp. 107-19.

A method of co-depositing tin and nickel, in roughly equal atomic proportions was described by PARKINSON in 1951 (*Jnl. Electrodepositors' Tech. Soc.*, vol. 27, pp. 129-51; *Nickel Bulletin*, 1951, vol. 23, No. 5, p. 101). The work covered in that paper was concerned with solutions containing stannous chloride, nickel chloride, ammonium bifluoride, and sodium fluoride, co-deposition of tin and nickel being dependent on the complexing action of the fluorides, whereby the deposition potentials of the tin and the nickel were brought sufficiently close to one another to make possible deposition of an alloy. The author then described the essential features of the process only in sufficient detail to permit an understanding of its technology: the later paper which is the subject of this abstract reports some of the detailed research carried out in developing the process.

In the absence of fluorides satisfactory deposition of a tin-nickel alloy is not possible from chloride solutions. Evidence is adduced by the authors that the fluorine complexes both the tin ions and the nickel ions. The constancy of composition which is a feature of coatings deposited from the electrolyte recommended suggests that deposition is associated with the presence, in the electrolyte, of a tin-nickel-fluorine complex. Complex-ion formation was studied by means of cathode-potential/current-density curves. The influence of variation in composition of the bath, and operating conditions, on the composition of the deposit, is reviewed, and information is given on the conductivity and throwing power of the electrolyte. Examination of the X-ray structure of alloy deposits covering a wide range of composition indicates that bright deposits are characterized by a preferred orientation of the crystallites.

From solutions of the type considered, tin-nickel alloys can be obtained only by operating at temperatures above 45°C.: the optimum temperature was found to be 65°C. The metastable structure of the deposits is believed to be explained by the high activation energy of the discharging cations, resulting from the high temperature of operation.

Tin-nickel deposits produced from baths of this type normally contain about 65 per cent. tin, 35 per cent. nickel. They have a pleasing colour, with a faint pink tint, and show a good degree of resistance to atmospheric tarnishing. Industrial application of the process is stated to have been basically satisfactory, but the brightness of deposits at present producible is not quite sufficient to permit dispensing with final finishing, and some trouble has been experienced with pitting. Further research is now directed towards finding addition media which will enhance brightness and eliminate pitting, and to improvements in means for circulation and filtration of the electrolyte.

Electroplating on Aluminium: Laboratory Studies of New Method

J. EDWARDS and C. J. SWANSON: 'A Laboratory Study of a Commercial Method of Electroplating Aluminium.' *Trans. Inst. Metal Finishing*, 1953, vol. 29, Advance Copy 5, Apr., 1953; 11 pp.

The authors report a laboratory investigation of the electrolytic method of plating on aluminium described by WALLBANK (*Jnl. Electrodepositors' Tech. Soc.*, 1952, vol. 28, Advance Copy 7: *Nickel Bulletin*, 1952, vol. 25, No. 6, p. 148).

Most of the work was carried out on aluminium of commercial purity: some tests were also made with five types of aluminium-base alloy. The initial experiments were made with a standard cycle of operations which had been found to give satisfactory results in commercial practice. These involved (i) trichlorethylene degreasing, (ii) cathodic cleaning, (iii) water rinsing, (iv) acid dipping, (v) water rinsing, (vi) cathodic cleaning, (vii) zinc plating, (viii) brass

plating, (ix) double water rinsing, (x) nickel plating, (xi) water rinsing, (xii) hot-water rinsing and drying, (xiii) stoving at about 220°C. Full details are given of the compositions of the solutions used for the various treatments. Some of the specimens were subsequently polished and chromium plated.

Qualitative adhesion tests gave satisfactory results on all the specimens except the aluminium-magnesium alloys.

The next stage of the investigation covered a study of the effect of varying the time of acid-dip, second cleaning, zinc-plating, and brass-plating treatments. Neither in the specimens plated under standard conditions nor in the modified conditions studied was an adherent coating obtained without the final heat-treatment. It is therefore concluded that when the standard procedure is used the stoving operation is essential for strong adhesion.

In an attempt to dispense with stoving, various modifications of procedure were made, on lines suggested by consideration of the immersion-zincate treatment used for plating of aluminium-base materials. It was found that a dip in sodium-hydroxide solution prior to the electrolytic zinc flash, or the replacement of the cleaning pre-treatment by that used in the immersion-zincate method, was unsuccessful. When, however, the electrolytic zinc deposit was applied to a surface which had previously been stripped of a zincate immersion deposit, strong adhesion was obtained without stoving. The modified process thus developed comprised the standard procedure referred to above, with the omission of stage xiii (stoving), and the insertion, between stages vi and vii (second cathodic cleaning and electrolytic zinc flashing), of the following four operations: immersion in sodium-zincate solution at room temperature, water rinsing, stripping of zincate immersion deposit by immersion in nitric acid, and water rinsing.

The achievement of strong adhesion without the need for stoving gives also the possibility of plating heat-treatable aluminium alloys, the properties of which might be impaired by a stoving operation. It also appears that the advantages of using bright nickel might be obtained when plating on aluminium. The modified process proved to be applicable to the 3·5 per cent. magnesium alloy, as well as to those types which could be treated by the standard method. It also gave good adhesion on an alloy containing 7 per cent. of magnesium, but only after stoving.

In the modified and the standard processes the sequence of operations is the same (apart from stoving) from the stage of entry of the specimens into the zinc-plating solution. It is therefore clear that at this stage there must be a difference in the surface condition of the basis material as treated by the two different methods, since in the one case strong adhesion is obtained after completion of the plating cycle, whereas in the other stoving treatment is necessary. The difference is also apparent in that when the non-electrolytic method is employed a double zincate treatment is frequently more successful than a single treatment.

The authors suggest a tentative explanation for these effects, the general line of argument being in accord with the views put forward by G. L. J. BAILEY (*Jnl. Electrodepositors' Tech. Soc.*, 1951, vol. 27, p. 233), except for some change in emphasis.

'Kanigen' Nickel-Plating Process

'New Plating Process Saves Nickel.' *Steel*, 1953, vol. 132, Mar. 23, p. 52.

This note summarizes claims made for the properties of nickel coatings deposited by simple immersion from a phosphide type of solution. No details are given of the composition of the bath, but it is stated that the process has been commercially developed from research carried out at the National Bureau of Standards by Brenner and others.

It is claimed that a non-porous, uniform coating can be deposited, on a wide range of materials, and that the process is particularly well suited for use on parts of intricate shape. The coatings have, however, low ductility, and are not recommended for parts subject to flexing. See also fuller description of the Kanigen process in *Metal Finishing*, 1953, vol. 51, Apr., pp. 96-7.

Determination of Boric Acid in Nickel-Plating Baths

E. J. SERFASS, R. B. FREEMAN and E. PRITCHARD: 'The Determination of Boric Acid in Nickel Plating Baths.' *Plating*, 1953, vol. 40, Jan., pp. 59-62.

Report issued under the aegis of American Electroplaters' Society Research Project No. 2, as the first of a series dealing with determination of constituents occurring in macro amounts in plating solutions.

The standard method at present used involves titration of boric acid in the presence of nickel, by standard alkali. The addition of mannitol is necessary in order to transform the weak tribasic boric acid into a stronger monobasic acid which can be titrated with standard alkali.

$$C_6H_{14}O_6 + H_3BO_3 \rightarrow C_6H_{12}O_6 \cdot BOH + 2H_2O$$

The indicator generally used for this titration is a mixture of bromthymol blue and bromcresol purple. Phenolphthalein is the recommended indicator for the determination of boric acid as a monobasic acid, but cannot be used in nickel solutions, due to the hydrolysis of nickel, to form $Ni(OH)_2$. An improved method for the determination of boric acid by eliminating the interference of nickel is desirable.

Three procedures may be used for removing nickel, and thus preventing interference: (1) deposition by electrolysis, (2) precipitation, and (3) formation of complex nickel compounds. Elimination by precipitation was selected as being the simplest of these methods. Separation of the nickel by precipitating the ferrocyanide was found to be satisfactory, and filtration of the nickel ferrocyanide proved unnecessary. The improved method (of which full details are given in this report) consists in (1) precipitation of the nickel as ferrocyanide, (2) titration with standard alkali to a bromcresol purple endpoint, to determine any mineral acid which may be present, (3) conversion of the weak tribasic boric acid to a strong monobasic acid, and (4) titration with standard alkali to a phenolphthalein endpoint.

NON-FERROUS ALLOYS

Hall Effect in Nickel and Nickel-Copper Alloys

A. I. SCHINDLER and E. M. PUGH: 'The Hall Effect of Copper-Nickel Alloys.' *Physical Rev.*, 1953, vol. 89, Jan. 1, pp. 295-8.

Report of measurements of ordinary and extraordinary Hall effects at room temperature, for nickel, copper, and alloys containing 10, 20, 30, 40, 50, 60, 80, 90 and 95 per cent. of copper. Each sample was measured in the un-annealed state and after annealing for two hours.

Nickel-Chromium-Aluminium Alloys: Constitution

A. TAYLOR and R. W. FLOYD: 'The Constitution of Nickel-Rich Alloys of the Nickel-Chromium-Aluminium System.' *Jnl. Inst. Metals*, 1952-53, vol. 81, May, pp. 451-64.

This paper reports one section of an extensive investigation of nickel-base alloys containing chromium, titanium and aluminium. (Earlier papers have dealt with the nickel-chromium-titanium and nickel-titanium-aluminium systems: *ibid.*, 1951-52, vol. 80, pp. 577-87; 1952-53, vol. 81, pp. 25-32: *Nickel Bulletin*, 1952, vol. 25, Nos. 8-9, p. 202 and 1952, vol. 25, No. 11, p. 262.) Revisions of the binary nickel-chromium and nickel-aluminium systems which have been described in the previous papers have been incorporated in the work now reported.

As in the case of materials for the other sections of the investigation, all the alloys are synthesized by melting together the component metals in charges of about 50 g. The raw materials used were Mond nickel pellet, chromium from Murex, Ltd., and high-purity aluminium from The British Aluminium Company. Equilibrium relationships in alloys containing more than 50-at. per cent. nickel were studied over the temperature range 750°-1150°C. The first stage of the research involved study of the X-ray-diffraction patterns of powder samples slowly cooled from 900°C. over a period of two weeks. Subsequently, X-ray and microscopic techniques were employed on isothermally heat-treated samples.

From their experimental data, which are fully recorded, the authors draw the following conclusions:—

'The part of the nickel-chromium-aluminium system defined by the composition triangle Ni-Cr-NiAl is in essence a self-contained ternary eutectic system in which the participating solid phases are the solid solution based on nickel (γ), NiAl(β), and chromium (α). The temperature of the ternary eutectic is 1320° $\pm$10°C. The solidification of low-chromium alloys containing 20-30 at.-per cent. aluminium is complicated by the formation of the γ' phase, based on Ni_3Al, viâ a peritectic reaction.

'As the temperature falls, the γ and β phase fields, which are extensive at the solidus, contract towards the nickel corner and NiAl, respectively. At the same time, the γ' phase field extends in the direction of the nickel-chromium edge of the composition triangle, chromium atoms replacing both nickel and aluminium in the Ni_3Al structure. From the solidus to a temperature in the neighbourhood of 1000°C. the γ and β phases are in equilibrium with each other, the two-phase field diminishing in extent until it disappears in a four-phase plane representing the isothermal reaction

$$\beta+\gamma \rightleftarrows \alpha+\gamma'$$

The two-phase ($\alpha+\gamma'$) field which is formed as a result of this reaction widens as the temperature falls.

'The face-centred cubic γ and γ' phases have separate identities throughout the temperature range explored. The equilibrium between them is such that their lattice parameters in some of the ($\gamma+\gamma'$) alloys are almost identical, giving rise to microstructures in which the precipitated γ' assumes a cubic form.

'Although specific-heat and resistivity measurements have shown that the γ phase is ordered below 540°C. at the Ni_3Cr composition, superlattice lines cannot be seen in Debye-Scherrer photographs of this alloy. However, they show clearly in diffraction patterns of similar alloys containing 5 at.-per cent. aluminium after slow cooling to room temperature. Diffraction patterns of alloys quenched from 1150°C. show that ordering persists to this temperature when the aluminium content is more than 10 at.-per cent.'

Constitution of Platinum-Copper-Nickel Alloys

V. G. KUZNETSOV: 'X-Ray Investigation of Alloys of the System Platinum-Copper-Nickel.'
Izvest. Sekt. Fiziko-Khim. Anal., 1948, vol. 16, No. 4, pp. 150-67; *Metallurgical Abstracts*, 1953, vol. 20, Feb., p. 401.

Report of study of ternary alloys containing 50, 60, 80 and 90 at.- per cent. platinum, and of binary platinum-copper alloys containing 24·97, 50·22, 60·73 and 73·99 at.-per cent. platinum. The following results are quoted in Metallurgical Abstracts, for the platinum-copper-nickel series: 'Ternary alloys heated at 950°C. or higher formed a continuous series of solid solutions with a face-centred cubic structure. The compound PtCu, having a rhombohedral lattice, separated out on slow cooling of the ternary alloy containing platinum 50, copper 45, nickel 5, at.-per cent. Measurements of the lattice of the ternary alloys showed deviations from Vegard's law: in this case the lattice expanded. It was further observed that the combined action of copper and nickel in this respect affected the expansion of the lattice more than the same number of atoms of one of these elements alone.'

Pickling of Metals and Alloys

O. VOGEL, H. VOGEL *et al.*: 'Handbook of Metal Pickling. Vol. I. Non-Ferrous Metals.' 2nd. revised, edn., 1951. Published by Verlag Chemie, Weinheim/Bergstrasse. pp. xv+410. Price 49.20 D.M.

The book is a comprehensive treatise on all aspects of the pickling and etching of non-ferrous metals and alloys. In section one, a history of development is followed by discussion of equipment, ventilation, and other details requiring consideration in connexion with both laboratory and large-scale operations. Disposal of waste liquors and health hazards are also

dealt with, with recommendations on preventive and remedial treatments.

The first chapter of section two covers operations involved in preparation of materials before etching: it includes information on bright heat-treatment and on degreasing methods. Later chapters are concerned with pickling and etching treatments suitable for individual groups of metals and alloys: the information on nickel and its alloys (pp. 265-74) covers nickel, Monel and Inconel, and nickel silvers are dealt with in the copper-base group (pp. 225-31). An additional chapter is devoted to treatment of antique metallic materials. Extensive references to the literature are given in all chapters.

NICKEL-IRON ALLOYS

Measurement of Magnetostriction in Nickel and Nickel-Iron Alloy

R. M. BOZORTH and R. W. HAMMING: 'Measurement of Magnetostriction in Single Crystals.'
Physical Rev., 1953, vol. 89, Feb. 15, pp. 865-9.

The authors describe simplified procedure for determining the five magnetostriction constants of a single crystal of a ferromagnetic cubic crystal. The method is applied to a crystal of a 78 per cent. nickel nickel-iron alloy, to ascertain the magnetostriction associated with spontaneous magnetization in the [111] direction, a quantity important in relation to the 'Permalloy problem'. The constants for a single crystal of nickel were also determined.

See also—

Magnetic Crystal Anisotropy and Magnetostriction in Nickel-Iron Alloys

R. M. BOZORTH and J. G. WALKER: 'Magnetic Crystal Anisotropy and Magnetostriction of Iron-Nickel Alloys.' *Physical Rev.*, 1953, vol. 89, Feb. 1, pp. 624-8.

In any theory of the magnetic properties of nickel-iron alloys account must be taken of the magnetic crystal anisotropy and the magnetostriction at saturation in different crystallographic directions. In this paper some reference is made to relevant work by Grabbe and by Lichtenberger, and an account is given of experiments in which magnetic crystal anisotropy and magnetostriction in various crystallographic directions were determined on the same specimens, subjected to different rates of cooling after annealing. The alloys used ranged from 35 to 90 per cent. nickel.

The results showed that cooling rate has an important influence on anisotropy for compositions near $FeNi_3$, where atomic ordering occurs, and that there is a definite, though smaller, effect of cooling rate on magnetostriction.

The findings are discussed in relation to the 'Permalloy problem', which is to explain the peculiar magnetic properties of iron-nickel alloys having a face-centred cubic structure, especially the high permeabilities of commercial alloys containing about 78 per cent. of nickel, and their variation with heat-treatment. It is pointed out that when magnetization proceeds by domain-boundary displacement, magnetization gives rise to a minimum of strain energy when the magnetostriction is zero in the direction of easy magnetization. In pure quenched alloys this occurs at 79-80 per cent. of nickel, which is a composition close to that of the alloys of ordinary purity.

Nickel-Iron-Chromium Alloys for Magnetic Shunts

H. MASUMOTO, Y. SHIRAKAWA and T. OHARA: 'The Magnetic Shunt Characteristics of Iron-Nickel-Chromium Alloys and M.S. Alloy.'
Sci. Reports, Research Inst., Tôhoku Univ., 1952, A, vol. 4, pp. 237-45; *Metallurgical Abstracts*, 1953, vol. 20, Mar., p. 466.

The authors record examination of the magnetic properties of alloys containing nickel 30-60, chromium 1-18, per cent., approx., balance iron, over the interval $-50°$ to $+100°C$. Alloys within this range of composition have a magnetic transformation point at about 100°C.: they possess characteristics rendering them suitable for magnetic shunts. Magnetic permeability in a field of 400 oersted at 0°C. is about 5-20, the temperature coefficient of permeability is 0·003-0·040, and permeability varies approximately linearly with temperature. The name of 'M.S. alloy' is suggested for alloys of this type.

CAST IRON

Handbook of Defects in Castings

G. HENON (editor): 'Album of Foundry Defects. Vol. I. Grey Cast Iron and Miscellaneous Information.' Published by Editions Techniques des Industries de la Fonderie, Paris, 1952; 196 pp. Price 2,650 French francs.

The work on which this handbook is based was carried out under the auspices of the International Committee of Foundry Technical Associations. The volume is uniform in size with the book published in 1949 by the Institute of British Foundrymen. It is concerned almost solely with defects liable to occur in cast iron, and in presenting the information relevant to that aspect of the subject it also provides a useful glossary of terms used in iron foundries. Photographs and diagrams give clear indications of the nature and appearance of the respective defects discussed; possible causes are reviewed, and remedies are suggested. The notes and illustrations of individual defects are preceded by a chapter showing the classification of defects according to which the material in the album is grouped. This classification was adopted in 1950 by the International Commission on Defects in Foundry Materials, and further developed by the editor of the present volume.

CONSTRUCTIONAL STEELS

Isothermal Formation of Bainite in Nickel-Alloy Steels: Effects of High-Temperature Heating

S. A. COTTRELL and T. KO: 'Effects of High-Temperature Heating on the Isothermal Formation of Bainite.' *Jnl. Iron and Steel Inst.*, 1953, vol. 173, Mar., pp. 224-8.

The effects of high-temperature heating on the rate of the austenite-bainite transformation were investigated in steels of the following En types: 17 (1·5% Mn-Mo), 18 (1% Cr), 19 (low-Cr-Mo), 21 (3% Ni), 25 and 26 (2½% Ni Ni-Cr-Mo), and 31 (high-C, 1% Cr).

It was found that in nickel-chromium-molybdenum steels the incubation period is shortened and the rate of transformation is increased by austenitizing at a high temperature, but these effects were not observed in the other steels examined. In the nickel-alloy steels the effect can be removed by re-austenitizing at a lower temperature.

Decomposition of Austenite in Low-Alloy Nickel-Molybdenum Steel Weld Metal

E. F. BAILEY and W. J. HARRIS: 'Decomposition of Austenite in Low-Carbon, Ni, Mo Steel Weld Metal.' *Welding Jnl.*, 1953, vol. 32, Mar., pp. 134S-38S.

Isothermal-decomposition studies were made on small sections cut from a weld pad of the following composition: carbon 0·08, silicon 0·45, manganese 1·05, nickel 2·00, molybdenum 0·75, chromium 0·25, nitrogen 0·25, per cent. The TTT diagram showed three reaction ranges: upper and intermediate types of nucleation and growth reactions with no eutectoid microstructure, and a martensite reaction at lower temperatures. The influence of decomposition during cooling to an isotherm, on isothermal decomposition, is discussed. A continuous-cooling diagram obtained by dilatometry indicated initiation of the transformations along horizontal temperature shelves, except in the regions of partial suppression. A completely martensitic structure was obtained only at water-quenching rates.

Temper-Brittleness: Critical Review of the Literature and Experimental Study

B. C. WOODFINE: 'Temper-brittleness: A Critical Review of the Literature.' *Jnl. Iron and Steel Inst.*, 1953, vol. 173, Mar., pp. 229-40.

A critical survey is made of published work on temper-brittleness up to 1952, supported by a *bibliography* containing 77 items. Individual sections comprise a historical introduction, and summaries of the literature relating to the following aspects of the subject: effect of temper-brittleness on mechanical and physical properties; measurement of temper-embrittlement and temper-brittleness; metallographic investigations; fractographic results; effect of time and temperature of heating; effect of composition; effect of microstructure; theories which have been advanced to explain temper-brittleness.

From the information available the author draws the following general conclusions:

'Although the outlines of the problem of temper-brittleness are clear, many aspects require further investigation and clarification before a satisfactory theory can be agreed upon.

'There is a danger that the problem may become obscured by differences in nomenclature. It has been suggested, for example, that any embrittlement (as indicated by an increase in the transition temperature of the fracture-energy/testing-temperature curve) which occurs on tempering should be called temper-embrittlement, irrespective of the cause or nature of the changes involved. This might well lead to confusion, and a better approach would appear to be to determine the nature and characteristics of the embrittlement in, say, nickel-chromium steels (to which the name temper-brittleness was originally given) and then to restrict the term temper-brittleness to those cases where the same features are observed.

'The facts which may be regarded as established by previous work are as follows:—

'(1) The embrittlement occurs in steels containing chromium, manganese, tungsten, molybdenum and nickel, although steels which contain only nickel do not appear to be susceptible, and molybdenum in combination with the other elements may reduce the susceptibility. Sulphur and aluminium do not appear to affect temper-brittleness, and it does not occur in plain carbon steels with less than 0·6 per cent. manganese.

'(2) Temper-brittleness displaces the low-temperature portion of the fracture-energy/testing-temperature curve to higher testing temperatures, but does not appear to affect the other mechanical properties of the steels. It has very little effect, if any, on the physical properties of the susceptible steels.

'(3) Temper-brittleness cannot be detected by any of the normal etching reagents, although it is revealed by etchants based on picric acid and containing small traces of surface-active compounds.

'(4) Temper-brittleness occurs in steels with a tempered martensitic microstructure, either on slow cooling from 650°C. or on reheating in the range 350°-650°C. It reaches a maximum after treatment between 475° and 525°C. Temper-brittleness does not occur to any appreciable extent above 600°C., and can be removed from a susceptible steel by quenching from about 650°C.

'The aspects of temper-brittleness which require further investigation include:

'(1) The effects of such alloying elements as phosphorus, nitrogen, titanium, cobalt, vanadium, and boron.

'(2) The effect of temper-brittleness on the low-temperature tensile properties of the susceptible steels.

'(3) The nature of the etch produced by the reagents which reveal temper-brittleness, and the relationship between the etch and the normal microstructure of the steels.

'(4) The possibility of a decrease in the temper-embrittlement with long-time treatments above 500°C. and also the maximum temperature at which temper-brittleness can occur.

'(5) The occurrence of temper-brittleness in microstructures other than tempered martensite, and the effect of such variables as grain size on the development of the embrittlement.

'(6) The effect of temper-brittleness upon the nature of the ductile and brittle fractures in the susceptible steels.

'Until further investigations have settled some of the disputed features of temper-brittleness, a detailed consideration of the various theories proposed to explain the phenomenon would be of little value.'

See also—

B. C. WOODFINE: 'Some Aspects of Temper-Brittleness.' *ibid.*, pp. 240-55.

This paper reports experimental work carried out by the author of the review referred to in the preceding abstract.

Consideration of the results obtained by previous investigators led to the conclusion that, although the notched-bar impact test is open to serious objections regarding interpretation of results, it is the most suitable one for quantitative study of temper-brittleness. It was therefore used for the greater part of the work recorded in this paper. The theory of the notched-bar test is examined, and an attempt is made to relate it to the fundamental flow and fracture properties of the steels examined, and thus to explain the effect of temper-brittleness upon these properties. The compositions of the steels used are shown in the table below.

In view of the difficulty of interpreting the results of continuous-cooling experiments, temper-brittleness was produced entirely by isothermal treatments; all the specimens were water-quenched from the tempering and embrittling temperatures.

In measuring temper-brittleness, the transition temperature was defined as the lowest testing temperature at which the fracture of the test bar was 100 per cent. ductile. The theoretical basis of this definition is discussed in the section relating to the theory of the notched-bar impact test. From the practical point of view, it has the advantage that the transition temperature can be fairly accurately determined by visual inspection of the fractured surfaces of the test bars.

Temper-embrittlement was taken as the relative displacement of the transition temperature of the fracture-energy/testing-temperature curves. Full details are given of impact-test conditions and a drawing is shown of apparatus used for sub-zero tensile tests.

Earlier work had shown that temper-brittleness could be detected by the use of an ethereal solution of picric acid containing a surface-active compound, cetyl tri-methyl ammonium bromide (CTAB). Experiments by the present author indicated that a saturated solution of picric acid in distilled water would produce exactly the same effect as the CTAB reagent, and this simpler solution was adopted as the standard reagent. Particulars are given of the electron-microscope technique employed, and typical electron micrographs are shown. Detailed experimental data are recorded, including results of determination of the effect of time and temperature on development of temper-brittleness, influence of microstructure on susceptibility, effect of austenitizing conditions, effect of titanium and aluminium additions, and occurrence of temper-brittleness in the nickel-phosphorus steel (the last of the series shown in the table). From the data thus assembled, the following conclusions are drawn:

'The picric-acid etching reagent produces grooves at both the austenite and ferrite boundaries, and does not reveal a grain-boundary precipitate.

'Brittle fracture occurs in tempered specimens by cleavage across the ferrite grains, and in temper-embrittled specimens by failure along the previously existing austenite boundaries. Temper-brittleness reduces the brittle-fracture stress of a susceptible steel without affecting the yield stress or the strain hardening.

'Temper-embrittlement at 550° and 575°C. in a nickel-chromium steel reached a maximum, and then decreased, as the time of tempering increased. Temper-brittleness takes place in a nickel-chromium steel up to the Ae_1 temperature. Prolonged tempering of a nickel-chromium steel at 650°C. reduced the subsequent temper-embrittlement.

'Temper-embrittlement occurs in isothermally transformed microstructures, although the extent of the temper-embrittlement is less than in the corresponding tempered martensitic structure.

C %	Mn %	P %	S %	Si %	Ni %	Cr %	Al %	Ti %	N %
0·41	0·38	0·030	0·033	0·19	3·33	1·22	nil	nil	0·0102
0·35	0·38	0·029	0·031	0·30	3·29	1·06	0·12	0·47	0·0040
0·35	0·38	0·031	0·034	0·32	3·21	1·04	0·29	1·50	0·0051
0·36	0·45	0·032	0·032	0·42	0·30	0·12	0·58	3·20	0·0020
0·51	0·66	0·031	0·030	0·28	3·24	1·20	0·95	nil	0·0252
0·51	0·38	0·014	0·013	0·20	0·06	2·96			
0·33	0·59	0·037	0·031	0·27	2·92	0·87			
0·28	0·08	0·010	0·017	0·26	3·00	tr.			
0·27	0·08	0·134	0·017	0·25	2·94	tr.			

'Increasing the grain size of a susceptible steel increases both the amount of subsequent temper-embrittlement and the rate of embrittlement. With a constant grain size, neither the austenitizing time nor the quenching temperature affects the subsequent development of temper-brittleness.

'The addition of titanium to a nickel-chromium steel did not prevent temper-brittleness from occurring, but appeared to increase the temper-embrittlement. A similar effect was found for aluminium.

'A 3 per cent. nickel steel was only very slightly susceptible to temper-embrittlement, but a similar steel containing 0·1 per cent. of phosphorus was appreciably embrittled by the same treatment.

'Temper-brittleness is not the result of a simple precipitation of compounds from solution in the ferrite: it is suggested that it can be explained by the segregation of solute atoms to the grain boundaries in both the austenite and the ferrite.'

Nickel-Alloy Steels in Gear Components

'Machining the Wilson Epicyclic Gearbox.' *Engineering*, 1953, vol. 165, Mar. 20, pp. 363-5.

A detailed and well illustrated description is given of machining operations carried out in production of the Wilson epicyclic gearbox, which is made, in two patterns and two sizes, by Guy Motors, Ltd., for fitting to their commercial vehicles. The production of the gearbox involves machining of numerous components to precision limits, and in varying quantities. The principal components are magnesium-alloy castings or steel drop forgings: for machining the former high-speed steel tools are used: for the steels tungsten-carbide tools are employed. In a review of the materials standard for the respective parts, it is noted that planet wheels (of which there are three to each gear assembly) are made from drop forgings of 5 per cent. nickel case-hardening steel, that the brake band is a drop forging of 3½ per cent. nickel oil-hardening steel, and that the driving shaft is made from a forging of 65-ton nickel-chromium steel.

Fatigue Strength of Nickel-Alloy Steels: Influence of Testing Frequency

T. WYSS: 'Influence of Testing Frequency on the Fatigue Strength of Steels and Light Alloys.' *A.S.T.M. Bulletin*, 1953, No.188, Feb., pp.31-4; T.P.23.

Fatigue strength for one million cycles was determined for various types of carbon and alloy steels, at 350 and 10,500 cycles per minute, and for various light alloys, at 350 and 8,000 cycles per minute. The tests were carried out under fluctuating axial tensile stress, and the minimum stress was zero. For low-speed tests an Amsler 10-ton hydraulic Universal Testing Machine was used; high-speed tests were made on an Amsler Vibrophore. Both machines are described and illustrated, and a diagram is shown of the test specimen employed. The materials tested included a 3 per cent. nickel steel, and three specimens of 3-4 per cent. nickel nickel-chromium steel, one of which also contained a small percentage of molybdenum.

At the higher testing frequencies employed, carbon steels showed up to 7 per cent. higher fatigue strength than at the lower frequencies; in alloy steels the increase was up to only 3 per cent. In light alloys practically no difference was observed, except in a zinc-containing aluminium alloy. The differences in the results on the steels are considered to be so small as to justify utilization of the many advantages of high-speed testing, among which is the convenience of the shorter time for which it is necessary to maintain specimens at constant temperature when sub-zero or elevated-temperature testing is being carried out.

Estimation of Chromium in Steels, Nickel-base Alloys and Other Materials

A. A. R. WOOD: 'The Absorptiometric Determination of Chromium in Steels and Alloys.' *Analyst*, 1953, vol. 78, Jan., p. 54-60.

In the determination of chromium the colour due to iron in perchloric-acid solutions of dichromic acid is suppressed by addition of phosphoric acid, which also reduces the critical effect of the concentration of perchloric acid. Details are given in the paper of a spectrophotometric study of dichromic acid under these conditions.

An absorptiometric method is described which is specific for estimation of chromium in a wide variety of steels and alloys. The procedure is applicable to both residual amounts of chromium and the amounts used for intentional alloying: measurement of small amounts of chromium is made by a modified absorptiometric technique.

Results obtained in the application of the method to a wide variety of materials (including low-alloy nickel-containing steels, and nickel-chromium and nickel-chromium-iron alloys) show it to be rapid, and suitable for control analysis. The initial perchloric-phosphoric acid solutions may be used as the basis for determination of other elements in steel, particularly for estimation of manganese. Details of this phase of the work will be published later.

X-Ray Fluorescent Analysis of Nickel-Chromium-Iron Alloys and Alloy Steels

See abstract on p. 88.

HEAT- AND CORROSION-RESISTING ALLOYS

Gas Turbines: Progress Report

'Gas Turbine Progress Report, 1952.' *Trans. Amer. Soc. Mechanical Engineers*, 1953, vol. 75, Feb., pp. 121-234.

This report, prepared under the auspices of the Gas-Turbine Power Division of the American Society of Mechanical Engineers, comprises a series of papers tracing the history of various aspects of gas-turbine

development, and presenting a review of the current position. Introductory historical papers covering evolution of the concept of propulsion by gas turbine, its development, and its practical application, are followed by a consideration of materials used in various components, methods of cooling, and fuels. A subsequent paper deals with the effect of components, as regards design, arrangement and efficiency, on performance of the modern gas-turbine plant, and later contributions contain reviews of the position of gas turbines in automotive, railway, and marine applications, their place in stationary electric-generating plant, in various industrial fields, and in aviation. The papers contain a large amount of correlated information, on progress in many countries, and are supported, in most cases, by *bibliographies* giving reference to further information on the respective aspects of the subject.

Nickel-containing Materials in Industrial Gas Turbine

'750 kW Open-Cycle Gas Turbine.' *Engineer*, 1953, vol. 195, Mar. 13, pp. 381-3.

Description of 750 kW/1000 kW 'T.A.' gas turbine now in series production at the Lincoln works of Ruston and Hornsby, Ltd. It is based on the Company's original 750 kW. unit, which has successfully undergone an extensive development programme. When running with its heat exchanger the turbine is rated at 750 kW, and in a non-recuperative form the rating is 1000 kW maximum.

The 'T.A.' turbines are destined for a range of applications extending from base load generation of power (in some cases with waste-heat recovery) to standby and portable duties. This article gives full details of design, and includes information on the materials used for many of the major components. It is noted that all turbine rotor blades are machined from Nimonic 80A, and that stator blades are of Nimonic 80. The intermediate compressor rotor and stator blades are machined from bar of En 56A high-chromium steel, but the inlet and exit stators are precision-cast by the lost-wax process, in 22-12 chromium-nickel steel. Of the 'T.A.' units already allocated, three are for the Air Ministry. These are arranged to burn any kind of distillate fuel oil, and to be coupled to a 3·3 kV, 50-cycle alternator. Each set is mounted on an integral base, and can be broken into three separate components, each complete with rollers, for wheeling into an aircraft and flying to site. A unit ordered by the Kuwait Oil Company is to drive a crude-oil pump, and is to burn natural gas: it will be installed in the crude-oil gathering centre at Kuwait, for pumping oil to the coast, where it is loaded onto tankers. Another standard model, without heat exchanger, coupled to an 11 kV alternator and exhausting into a waste-heat boiler, is being supplied to the Crittall Manufacturing Company. Coal-tar fuel will be used for this installation, and the exhaust gas from the turbine is to be ducted to a high-pressure hot-water boiler, the output of which will be used for process work and space heating.

Chromium-Nickel Steel Pressure Vessels: A.S.M.E. Code Ruling

'A.S.M.E. Boiler Code Case Interpretations.' *Mechanical Engineering*, 1953, vol. 75, Mar., pp. 251-3.

The Boiler Code Committee of the American Society of Mechanical Engineers meets monthly to consider cases in which users have found difficulty in interpreting the A.S.M.E. Code. Details of the rulings given in such cases are published in *Mechanical Engineering*. A decision recently given affects the use of chromium-nickel steels, as such or alloyed with niobium, titanium, or molybdenum, in the construction of unfired pressure vessels. The answer was affirmative, subject to certain specified conditions of composition, testing, heat-treatment, thickness-, temperature-, and working-stress-limitations, and with certain precautions in fabrication. Details are given in the Case Ruling. A non-mandatory appendix to the decision makes suggestions on selection and treatment of austenitic chromium-nickel steel for this purpose, including proposed ranges of temperature for heat-treatment of pressure vessels made from various grades of steel.

Influence of Chemical Composition on High-Temperature Strength of Chromium-Cobalt-Nickel-Iron-base Alloys

E. E. REYNOLDS, J. W. FREEMAN and A. E. WHITE: 'Influence of Chemical Composition on Rupture Properties at 1200°F. of Forged Chromium-Cobalt-Nickel-Iron-base Alloys in Solution-Treated and Aged Condition.' *Nat. Advisory Committee for Aeronautics, Report* 1058, 1951; 60 pp.

Supersedes N.A.C.A. Technical Note 2449, 1951; see abstract in *Nickel Bulletin*, 1952, vol. 25, No. 3, pp. 73-4.

Nickel-Chromium-Aluminium Alloys: Constitution

See abstract on p. 80.

Nickel-containing Titanium-Carbide-base Materials

R. KIEFFER and F. KÖLBL: 'The Development and Properties of Heat- and Scale-Resisting Hard Alloys based on Titanium Carbide and containing Nickel, Cobalt, and Chromium as Binding Agents.' *Planseeberichte für Pulvermetallurgie*, 1952, vol. 1, Oct., pp. 17-35.

This paper gives a review of work done at the Versuchsanstalt der Metallwerk Plansee Ges. m.b.H. (Reutte, Tirol) and elsewhere, on materials of titanium-carbide base, with or without various metals as binders. Reference is given to publications which have reported this work over the period 1933 to 1951, and particular attention is directed to a series of materials developed by Metallwerk Plansee under the designation 'WZ' (warmfest under zunderfest=heat- and scale-resisting). These materials are produced by (1) sintering mixtures of carbide with nickel and/or cobalt (as binding media), with or without other types of metal and carbide, or (2) impregnation of the carbide base with molten metals. Attention is called to the properties of materials containing:

(*a*) titanium carbide 60, nickel 24, cobalt 8, chromium 8, per cent., which showed specially high resistance to oxidation, (*b*) titanium carbide 50, nickel 30, cobalt 10, chromium 10, per cent., which, at room temperature, showed a tensile strength of 75-85 kg./mm² (47·5-54 tons per sq. in.), and (*c*) titanium carbide 60, nickel 32, chromium 8, per cent., which was characterized by particularly high mechanical strength at temperatures of the order of 800°-1000°C. The physical and mechanical properties of many types of the carbide-base materials are tabulated and discussed, and their potential value in high-temperature applications is indicated.

Variation in Corrosion-Resistance of Niobium- and Titanium-Stabilized Austenitic Steels

G. W. WEEKS: 'Niobium- versus Titanium-Stabilized Steel.' *Metal Progress*, 1953, vol. 63, No. 2, pp. 116-118.

Further to discussion (*ibid.*, July and November, 1950) of the relative merits of niobium and titanium as stabilizers for chromium-nickel austenitic steels, the following experience is recorded from the works of De Havilland Aircraft Company, Ltd.

A special pickle is usually employed for removal of surface oxide and borax-type welding flux from oxyacetylene-welded stainless-steel pressings made from material about 0·035-in. thick. The pickle contains 12 per cent. of concentrated nitric acid and 3 per cent. of concentrated hydrochloric acid (both by weight), plus Hibitite (a proprietary inhibitor sold by Monsanto Chemicals, Ltd.), added in the proportion of 0·35 per cent. by weight to the weight of concentrated acid. Pickling is by immersion for one hour at 65°-75°C.

A tank of titanium-stabilized chromium-nickel steel used to hold the solution has given very long life, and titanium-stabilized steel parts have been satisfactorily treated in such a bath for many years. Violent corrosive attack has, however, occurred on niobium-stabilized steel when pickled under the same conditions. Cold-worked material appears to be specially prone to attack; heat-treatment at about 1900°F. (1036°C.) has been found to reduce susceptibility, but does not entirely eliminate it. Although the writer feels that the evidence is at present insufficient to attribute the difference in behaviour solely to the stabilizing agent, he considers that, in the light of statements made earlier on the impairment of oxidation-resistance by niobium, the influence of this element must be viewed as deleterious in relation to corrosion-resistance. The experience of others using titanium and niobium as stabilizers would be welcomed.

Stabilization of Welds in Austenitic Nickel-Chromium Steel

H. F. TREMLETT: 'Electrodes for Welding 18% Cr-8% Ni Steels.' *Welding*, 1953, vol. 21, Apr., pp. 136-44.

Austenitic welding electrodes stabilized with niobium usually have a niobium content of 1·0-1·4 per cent. The author of this paper describes experiments arising out of the examination of electrodes in which the niobium was at a markedly lower level, and which failed to pass the weld-decay-resistance approval test. The programme of tests was drawn up with a view to determining what would be a safe niobium content in the built-up weld-metal deposits, due consideration being given to the effect of the parent-steel composition on the actual welds used in the fabricated article.

As a preliminary to investigation of weld decay in the deposited metal, susceptibility to such deterioration in the parent steel is discussed. The nature of the phenomenon is described, and notes are given on weld-decay testing with the standard copper-sulphate/sulphuric-acid solution. An account is given of tests made, on parent austenitic chromium-nickel steel plate, to assess the influence of carbon content, temperature of heating, and time of exposure to the test solution, on the occurrence of intercrystalline breakdown. The mechanism of breakdown, as covered by the chromium-impoverishment theory of weld decay, is explained, and, taking this explanation as valid, the author outlines the methods which may be adopted for prevention of weld decay:—

(1) re-dissolving any carbide which has been precipitated, before the welded structure is put into service,
(2) causing chromium to diffuse from the centre of the grains towards the boundaries, to replace the chromium lost to the carbide,
(3) preventing migration of carbon to the boundaries (*a*) by ensuring a carbon content not exceeding that soluble in austenite at 650°C., which is believed to be less than 0·02 per cent., or (*b*) by combining all the carbon with some element other than chromium, so that the chromium remains unaffected by the varying solubility of the particular carbide,
(4) permitting only a very small amount of carbide to form at the boundaries. This requires a very low carbon content, of the order of 0·02-0·03 per cent.

The practicability of each of these methods is briefly discussed.

The next section of the paper deals with testing of electrodes; it includes a description of the method adopted as standard by the United Steel Companies, Ltd., with which the author is associated.

The final section contains an account of tests made (over a period of twelve years) to determine reliable data for assessing the minimum niobium-to-carbon ratio essential in the weld metal to ensure freedom from susceptibility to weld decay. In this connexion account must be taken of dilution from the titanium-bearing chromium-nickel steel parent plate, which is niobium-free but may be of higher carbon content than the undiluted weld metal. The experiments described permit determination of the desirable niobium range for undiluted weld metal deposits, taking into account the effect of titanium, nitrogen, and carbon from the plate, on the undiluted weld bead. The data cover parent steels at the 0·05-0·08 and 0·10 per cent. carbon levels, with 8-to-1 titanium-to-carbon ratio.

It is concluded that diluted weld beads of adequate weld-decay resistance will be obtained if electrodes for welding the general run of commercial titanium-

treated steels give an undiluted weld metal with a niobium content within the range 1·0-1·5 per cent. (preferably 1·2-1·4 per cent.) and a carbon content of 0·05-0·08 per cent.

Nickel-base Alloy for Pulp Digesters

'New Alloy Tried in Pulp Digester Resists Corrosion.' *Iron Age*, 1953, vol. 171, Mar. 26, pp. 148-50.

Notes on the properties of Hastelloy F, an alloy highly resistant to a wide range of acid and alkaline solutions, and suitable, *inter alia*, for use in both alkaline and acid pulping digesters, in either the liquid or the vapour phase. The composition of the alloy (not given in this article) has elsewhere been reported as iron 24, chromium 22, molybdenum 6, niobium-tantalum 2, carbon 0·05, nickel (balance) about 46 per cent. Broad claims made for the physical and mechanical properties of the alloy are as follows:

It is stronger, and about as tough, as austenitic stainless steels. It is sufficiently ductile for intricate cold-forming operations, but requires more power to deform it than do the austenitic stainless steels. Strength does not change significantly after furnace cooling from 1000°-2000°F. (540°-1095°C.). It loses some ductility when slowly cooled from 1600°-2000°F. (870°-1095°C.): ductility is lowest at 1300°-1600°F. (705°-870°C.), and severe hot-forming operations should not be attempted in this range. Density of Hastelloy F is comparable to that of other nickel-base alloys; electrical resistance is somewhat higher than that of 18-8 chromium-nickel steel, and the thermal-expansion properties are about 10 per cent. lower. The modulus of elasticity of the alloy approaches that of steel. Thermal conductivity has not yet been determined, but is expected to be similar to that of 18-8 steel. Hastelloy F can be welded by the metallic-arc, inert-gas-shielded arc, and resistance welding processes.

Welded Nickel Tubing for Transport of Caustic Soda

See abstract on p. 76.

Stainless and Heat-Resisting Steels Used in Russia

C. A. ZAPFFE: 'Russians Show Progress on Stainless and Heat-Resisting Steels.' *Iron Age*, 1953, vol. 171, Mar. 19, pp. 138-42.

Compositions of stainless and heat-resisting steels currently used in Russia are tabulated and are compared with those of steels employed for similar purposes in U.S.A. The comment is made that, in general, Russian stainless-steel technology bears the imprint of the 1930's. For example (1) their version of Type 316 stainless steel (molybdenum-containing 18-8) shows no signs of the adjustments of composition which have been introduced by steelmakers in other countries to prevent formation of ferrite and sigma; (2) there appears to be an absence, from Russian specifications, of free-machining grades of stainless steel, free-spinning modifications, stabilized grades other than straight 18-8, extra-low-carbon grades, and precipitation-hardening types.

Among other salient features of Russian steels are the following: tungsten is the addition mainly used to ensure strength at high temperatures; silicon is widely employed to secure scaling-resistance, and it is thought that some of the silicon-containing types represented may deserve attention; no cobalt-containing types of high-temperature alloy appear in the Russian lists; no significant percentages of molybdenum are shown in heat-resisting alloys.

High-alloy cast irons in production in Russia include both high-chromium and austenitic nickel-containing types.

The iron-base alloys containing both aluminium and chromium have received close study in Russia: typical compositions are listed. These alloys are designed primarily to secure scaling-resistance, but it is reported that some of them have useful mechanical properties at high temperatures.

The review is supported by references to eleven items of relevant literature.

Descaling of Stainless Steel by the Sodium-Hydride Process

N. L. EVANS: 'Descaling Stainless Steel Tube.' *Iron and Steel*, 1953, vol. 26, Apr., pp. 134-6.

The introduction of the sodium-hydride descaling process is generally recognized as having been one of the major recent technical and economic advances in processing of stainless steel. Speed of action, complete absence of attack on the metal (with consequent elimination of surface pitting), and saving of labour, combine to recommend its use. Sodium hydride is a powerful reducing agent, which, when dissolved in molten caustic soda, converts the oxides of most of the common metals to the metallic state. Scale produced by hot working and annealing is, after hydride treatment, usually left in the form of a loosely adhering powder which can be easily removed from the surface of the metal. The main operation is carried out in a bath in which the hydride concentration is not above 2 per cent., and no serious corrosion problems are encountered with regard to the plant used, virtually the whole of which is of mild steel. An added advantage is the absence of fumes which could constitute a health hazard or damage the fabric of the buildings.

This article gives a detailed and well illustrated description of the sodium-hydride descaling installation operating in the tube works of Messrs. Accles and Pollock, at Broadwell, which is designed to treat 25 tons of stainless steel tube per eight-hour shift. The actual descaling operation can be accomplished by immersion in the bath for only seven minutes, and a complete descaling cycle, including water quenching, a flash dip (if required) in a mixed-acid bath, and brushing in a lime-water bath, occupies only about 25 minutes. The sequence of operations is described by the author of this article, with general notes on layout of the plant, the sodium-hydride generators, working of the bath, subsequent processing of the treated tubes, and means adopted for protection of the operators from injury when preparing or working the baths.

Experience at the plant of Accles and Pollock indicates that the following advantages are gained by sodium-hydride descaling: (1) dies and plugs used in

tube drawing are found to have increased life, due, in part, to the lubricating properties of the thin layer of reduced scale which remains on the surface of the tubes; (2) there is no metal loss; (3) bigger batches of work can be dealt with; (4) the complete cycle of treatment involved in the output mentioned above requires only 24 operators, compared with 54 needed before this method was introduced. In addition to these direct savings, the quality of sodium-hydride descaled tubes is superior to that obtained by acid pickling. Further practical advantages are that when using this bath there is no danger of over-pickling; sodium-hydride concentration is not critical, and the plant required is not expensive. The sodium-hydride process is the subject of British Patent 557,530, but free user licences are granted by Imperial Chemical Industries, Ltd., to firms installing approved descaling plants.

Oxalic-Acid Electrolytic-Etch Test for Stainless Steels

M. A. STREICHER: 'Screening Stainless Steels from the 240-Hr. Nitric Acid Test by Electrolytic Etching in Oxalic Acid.' *A.S.T.M. Bulletin*, 1953, No. 188, Feb., pp. 35-8; T.P. 27.

The boiling 65 per cent. nitric-acid test (Huey test), which is much used in the U.S.A. for evaluation of susceptibility to intergranular corrosion and weld decay, involves exposure for 240 hours. This paper describes a rapid preliminary 'weeding-out' method, by which the number of steels which must be subjected to the long-time test can be minimized.

The test is as follows:—

Preparation of Sample: Rough polishing to 000 emery paper.

Etching: Anodic dissolution in 10 per cent. oxalic acid, for 1½ minutes, at 1 amp./sq. cm.

Examination: Microscopic examination of etched surface, at 250-500 magnification.

On steels which are free from intergranularly precipitated carbides a characteristic 'step' structure is produced by the above oxalic-acid treatment. Such steels, and 'dual-structure' steels, show low and constant corrosion rates in the standard nitric-acid test. These steels need not be subjected to the latter test. Steels which contain intergranularly precipitated carbides will, however, be heavily etched by the oxalic acid, producing 'ditches', which may surround whole grains. Such steels must be subjected to the nitric-acid test, to ascertain whether their degree of intergranular susceptibility is within that permissible by the standard test as maximum for the particular grade of steel concerned.

The test proposed is stated to be applicable to steels of the following types: 18-8, 18-8L, 18-8-Mo (wrought), and 18-8, 18-8-Mo and F.A. 20 nickel-chromium-copper-molybdenum (cast).

The author gives an account of other etching solutions which were studied in the course of the experimental work done in developing the oxalic-acid procedure. In some cases these were less discriminating than oxalic acid, in others they were applicable only to a much narrower range of steels. Oxalic acid was found to give the best etch structure (sharp and deep), in the shortest time, with a minimum of toxicity and inconvenience. The etching characteristics of a dilute sulphuric-acid solution (10 per cent.) most nearly approach those of oxalic acid.

Determination of Niobium, Tungsten and Tantalum in Stainless Steels

L. IKENBERRY, J. L. MARTIN and W. J. BOYER: 'Photometric Determination of Niobium, Tungsten and Tantalum in Stainless Steels.' *Analytical Chemistry*, 1953, vol. 25, Mar., p. 517. Abstract of paper presented at Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, March, 1953.

The increased use of ferroniobium containing appreciable amounts of tantalum (5 to 25 per cent.) has made it necessary to determine the tantalum as well as the niobium content of Type 347 stainless steel. These steels often contain varying amounts of tungsten which inadvertently get into the melt with the scrap. The separation of niobium, tungsten, and tantalum as a group, by the conventional hydrolysis scheme, is readily accomplished. Photometric procedures have been developed for determining these three elements after the group separation. The niobium and tungsten are determined by additive absorbencies, utilizing the colour reaction with hydroquinone, and the tantalum is determined by utilizing the colour reaction with pyrogallic acid. The method was found to be satisfactory when applied to the mixed oxides of niobium, tungsten, and tantalum after a preliminary group separation. This scheme can be applied also to the mixed oxides obtained from other materials, such as ores and slags.

X-Ray Fluorescent Analysis of Nickel-Chromium-Iron Alloys

E. GILLAM: 'Quantitative Analysis by X-Ray Fluorescence. Application to Alloy Systems.' *Metal Treatment*, 1953, vol. 20, Mar., pp. 99-102.

Quantitative determination of the elements in alloys, by the use of fluorescent X-rays emitted by them, can be carried out with a monitored Geiger counter spectrometer, but allowance must be made for the effects of absorption in the specimen and the mutual fluorescence between elements. This article describes the results of a systematic study of the possibilities and limitations of the method as a routine procedure for works analysis of alloy steels. The conclusions drawn are considered to be generally applicable to all metal mixtures and solutions.

From the results of original experiments reported, it is concluded that, with the usual powerful X-ray intensities, sufficiently accurate results can be obtained in a few minutes, provided that at least 10 per cent. of the element sought is present. It is believed that if a powerful rotating anode tube capable of being run at 100 mA were available it should be possible to determine an element present in amounts of about 1 per cent. Experiments recorded were carried out on chromium-iron alloy and on ternary alloys containing chromium and iron with varying amounts of nickel, with or without small amounts of molybdenum.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

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